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Summary of the 1982 National Performance Audit Program on Source Measurements

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A Summary of the 1982 National Performance Audit Program on Source Measurements

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16. ABSTRACT

In the spring and fall of 1982, the Quality Assurance Division conducted the National Audits for Stationary Source Test Methods. The audit materials consist of: a calibrated orifice for Method 5 (dry gas meter only), five simulated liquid samples each for Method 6 (SO_2) and Method 7 (NO_x), two coal samples for Method 19A, and a disposable gas cylinder for Method 3 (Orsat analyzer). Laboratories participating in the audits sent their data to the Source Branch and later received a written report comparing their results to EPA's.

In the Method 5 spring audit, the mean for all participants differed by 3.5% from the true (EPA) value. For the fall audit, the participants' mean was 8.5% from the true value. In the two Method 6 audits, the median values measured for all 10 samples differed by less than 2% from the true value, whereas the median values for all 10 samples used in the two Method 7 audits were within 3% of the true value.

In the two coal audits, the participants' accuracy on all four parameters was consistently better for the higher concentration samples than for the lower concentration samples. The mean values for sulfur, ash, and BTU content were within 5%, whereas the mean values for moisture differed by as much as 9%.

This was the first Method 3 audit conducted by the Quality Assurance Division. Each parameter had only one concentration. The median values of CO_2 and O_2 were within 12% of the true value, whereas the median values for CO were within 20% of the true value.

This report covers a period from January 1982 to December 1982.

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FOREWORD

Measurement and monitoring research efforts are designed to anticipate potential environmental problems, to support regulatory actions by developing an in-depth understanding of the nature and processes that impact health and the ecology, to provide innovative means of monitoring compliance with regulations, and to evaluate the effectiveness of health and environmental protection efforts through the monitoring of long-term trends. The Environmental Monitoring Systems Laboratory, Research Triangle Park, North Carolina, has responsibility for: assessment of environmental monitoring technology and systems; implementation of agency-wide quality assurance programs for air pollution measurement systems; and supplying technical support to other groups in the Agency, including the Office of Air, Noise and Radiation, the Office of Toxic Substances, and the Office of Enforcement.

The major task of this study was to report the results of the national quality assurance audit program for stationary source test methods. Audits were designed to estimate the minimal analytical and computational accuracy that can be expected with EPA Method 5 (dry gas meter only), Method 6 (sulfur dioxide), Method 7 (nitrogen oxides), Method 19 (coal), and Method 3 (carbon dioxide and oxygen). Statistical analysis was used to characterize the data.

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ABSTRACT

In the spring and fall of 1982, the Quality Assurance Division conducted the National Audits for Stationary Source Test Methods. The audit materials consist of: a calibrated orifice for Method 5 (dry gas meter only), five simulated liquid samples each for Method 6 (SO_2) and Method 7 (NO_x), two coal samples for Method 19A, and a disposable gas cylinder for Method 3 (Orsat analyzer). Laboratories participating in the audits sent their data to the Source Branch and later received a written report comparing their results to EPA's.

In the Method 5 spring audit, the mean for all participants differed by 3.5% from the true (EPA) value. For the fall audit, the participants' mean was 8.5% from the true value. In the two Method 6 audits, the median values measured for all 10 samples differed by less than 2% from the true value, whereas the median values for all 10 samples used in the two Method 7 audits were within 3% of the true value.

In the two coal audits, the participants' accuracy on all four parameters was consistently better for the higher concentration samples than for the lower concentration samples. The mean values for sulfur, ash, and BTU content were within 5%, whereas the mean values for moisture differed by as much as 9%.

This was the first Method 3 audit conducted by the Quality Assurance Division. Each parameter had only one concentration. The median values of CO_2 and O_2 were within 12% of the true value, whereas the median values for CO were within 20% of the true value.

This report covers a period from January 1982 to December 1982, and work was completed as of December 1982.

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We wish to express our appreciation to the laboratories that participated in our audits. Thanks also to the Standards Laboratory (EPA/EMSL/Performance Evaluation Branch) who did our Acceptance Testing on the audit samples. For providing the data systems for storing and evaluating the data, we wish to thank the programmers of the Data Management and Analysis Division/EMSL.

SECTION 1

INTRODUCTION

In 1977 the Environmental Monitoring Systems Laboratory (EMSL) of EPA established a performance audit program to evaluate the performance of companies that conduct compliance testing using EPA Reference Methods. The audits verify the analytical accuracy of EPA Reference Methods 3, 6, 7, and 19A and the calibration accuracy of the Method 5 control console (1). By participating in this free and voluntary program, testing companies are able to compare their performance to other laboratories conducting similar measurements.

In 1982 two audits each were conducted for Methods 5, 6, 7, and 19A, and one audit was conducted for Method 3. Each participating laboratory received an audit package consisting of the audit sample, a data card, instructions, and an envelope for returning the data to EPA. A label was also included for the Method 5 audits for returning the audit device and for the Method 3 audit for returning the Tedlar bag. Participants had 8 weeks to return data to EPA. At the end of this period all data received were statistically analyzed to determine the precision and accuracy obtained by the participants.

This report summarizes the results obtained in the 1982 source audits. Individual Method 3 results reported by each participant are contained in the appendices to this report.

SECTION 2

SUMMARY

In the spring and fall of 1982, EPA's Environmental Monitoring Systems Laboratory, Research Triangle Park, North Carolina, conducted National Quality Assurance Audits for Stationary Source Test Methods 5 (dry gas meter only), 6 (SO_2), 7 (NO_x), 19A (coal), and 3 (Orsat analyzer). Industrial laboratories, contractors, foreign countries, and local, state, and Federal agencies participated.

Two Method 5 audits were conducted in 1982. The overall results (no outliers removed) are summarized in Table 1. In the first audit, the mean for all participants was 3.5% from the true value and in the second audit it was 8.5%. After correcting for outliers, the means for 0382 and 0982 audits were 0.2% and 1.3% from the true value. The participants' performances were not statistically significantly different from previous audits (2,3).

TABLE 1. PARTICIPANTS' RESULTS FOR METHOD 5 AUDIT
(ALL DATA - NO OUTLIERS REMOVED)

Type of sample	Parameter	Audit date	No. of analyses	Mean (% from EPA values)	Median (% from EPA values)	Std. dev.
Orifice	Volume	0382	827	3.5	0.4	40.1
		0982	769	8.5	1.5	81.9

Table 2 presents the data (no outliers removed) from the two 1982 Method 6 audits. This audit procedure requires the participants to determine the sulfate content in five aqueous solutions using the titration procedure of Method 6. For each concentration in the 0282 audit, the means of the participants were 43% higher than the true value, but in contrast the median value differed by less than 1%. In the 0882 audit, the mean and median values of all concentrations differed by less than 2%. In both audits, 45% to 68% of the participants achieved an accuracy within 2% for 8 out of 10 samples. However, on the other two samples only 20% achieved this accuracy.

TABLE 2. PARTICIPANTS' RESULTS FOR METHOD 6 AND 7 AUDITS
ALL DATA (NO OUTLIERS REMOVED)

Type of sample	Parameter	Audit date	No. of analyses	EPA true value	Participant results		
					Mean	Median	Std. dev.
Aqueous Sulfate (Method 6)	SO_2	0282	115	381.3	555.2	381.9	1420.3
		0882	102	268.0	266.6	267.0	41.4
		0282	116	877.0	1256.1	882.3	3155.3
		0882	103	653.0	645.3	647.5	102.3
		0282	116	1410.8	2022.6	1423.4	5133.6
		0882	103	1740.0	1738.6	1710.0	280.7
		0282	116	1906.5	2721.4	1903.2	6863.1
		0882	103	1187.0	1165.4	1176.0	165.3
		0282	115	2325.9	3322.7	2320.0	8480.8
		0882	103	2224.0	2244.2	2210.0	354.6
Aqueous Nitrate (Method 7)	NO_x	0482	75	179.2	236.1	179.5	424.9
		1082	64	139.4	152.7	141.7	66.32
		0482	79	358.3	467.9	366.0	795.8
		1082	64	318.5	342.7	324.0	161.4
		0482	79	537.5	706.4	550.0	1227.8
		1082	64	477.8	534.3	485.8	267.4
		0482	78	736.6	952.3	747.4	1679.5
		1082	64	756.5	818.7	770.9	396.9
		0482	78	975.5	1274.5	986.6	2233.4
		1082	62	935.7	1034.6	948.0	475.4

Table 2 also presents the data (no outliers removed) from the two Method 7 audits in 1982. This audit procedure requires the participants to determine the nitrate content in five aqueous solutions. For each concentration in the 0482 audit, the means of the participants were 30% higher than the true value, but in contrast the median value differed by less than 3%. In the 1082 audit the means were 10% higher than the true value; however, the median values differed only by 2%. The participants' level of accuracy was consistent for all 5 samples in both audits; i.e., 40% were within 3% on all samples.

Table 3 summarizes the results of the two coal audits. Participants analyzed each coal sample in duplicate for percent sulfur, moisture, and ash, and for gross calorific value (BTU/lb). The means of the ash and BTU content were within 2% of the expected value on both concentrations. An accuracy of within 5% was achieved on the sulfur content. However, the mean value for moisture on the low-concentration was as high as 9% from the expected value.

TABLE 3. PARTICIPANTS' RESULTS FOR METHOD 19A COAL AUDIT
(ALL DATA - NO OUTLIERS REMOVED)

Type of Sample	Audit Date	Parameter	No. of analyses	EPA (true) value	Participants' results		
					Mean	Median	Std. dev.
Coal	0382	%S	73	1.30	1.25	1.24	0.17
	0982		81	1.22	1.28	1.25	0.31
	0382		73	2.85	2.85	2.83	0.39
	0982		81	3.22	3.17	3.20	0.22
	0382	%H_2O	73	0.96	1.03	1.10	0.28
	0982		82	2.11	2.29	2.36	0.55
	0382		73	1.73	1.74	1.81	0.35
	0982		82	2.48	2.57	2.66	0.54
	0382	%Ash	73	21.48	21.60	21.63	0.41
	0982		81	11.43	11.24	11.25	0.27
	0382		72	35.40	35.36	35.32	0.47
	0982		81	16.34	16.11	16.16	0.59
	0382	BTU/lb	71	9322.0	9409.5	9420.0	71.96
	0982		78	12277.0	12252.9	12293.0	184.07
	0382		71	11278.0	11408.6	11421.0	98.95
	0982		78	12932.0	12894.5	12915.0	159.75

The results of the first Method 3 audit conducted by the Quality Assurance Division (QAD) are summarized in Table 4. Participants analyzed the gas sample twice for percent carbon dioxide, oxygen, and carbon monoxide. The median values of carbon dioxide and oxygen were within 12% of the expected value. However, the median values for carbon monoxide differed as much as 20% from the expected value.

TABLE 4. PARTICIPANTS' RESULTS FOR METHOD 3 AUDIT
(ALL DATA - NO OUTLIERS REMOVED)

Type of sample	Audit date	Parameter	No. of analyses	Replicate	EPA (true) value	Participants' results		
						Mean	Median	Std. Dev.
Small cylinder	0682	$\%CO_2$	59	1	14.8	13.25	13.90	1.83
			56	2	14.8	13.29	13.75	1.80
		$\%O_2$	57	1	5.2	6.11	5.70	1.35
			54	2	5.2	6.20	5.80	1.42
		%CO	50	1	7.0	5.06	5.65	1.96
			47	2	7.0	4.96	5.70	2.05

SECTION 3

RECOMMENDATIONS

The Quality Assurance Division of the Environmental Monitoring Systems Laboratory maintains a repository of audit samples for EPA Methods 3, 6, 7, and for coal. These stable samples are available to any laboratory having a need for them, such as for training new personnel and conducting quality control checks of the laboratory. Since the expected values for these samples are included with the analysis instructions there is no requirement for the data to be returned to EPA. We recommend that participants make use of this sample repository to help improve their overall analytical skills.

SECTION 4

METHOD 5 DRY GAS METER AUDIT

In the Method 5 audit procedure, participants use a calibrated orifice to check the calibration of the dry gas meter in their EPA Method 5 control console (meter box). They insert the orifice in the Method 5 meter box, allow the box to warm up, and then make three 15-min volume measurements. Using Equation 5-1, they convert each of the three volumes to cubic meters at standard conditions, record them on the data card, and mail the orifice and the data card to EPA for statistical analysis.

In the spring audit (0382), 75% of the 165 laboratories that received the audit package returned data. In the fall audit (0982), 73% of the 164 laboratories returned data. These percentages are similar to those encountered in previous audits (2,3). Table 5, which classifies the participants into general categories, shows the number of participants who requested to participate in the Method 5 audit and the number who actually returned data.

TABLE 5. METHOD 5 AUDIT PARTICIPANTS

	No. requesting samples		No. returning data	
	0382	0982	0382	0982
Contractors	92	90	68	62
Industry	45	44	32	34
Foreign	3	3	3	3
Federal	3	4	2	3
State	15	16	14	12
Local	7	7	5	6
TOTAL	165	164	124	120

Figure 1, a cumulative histogram, shows the accuracy obtained by participants in the 0382 and 0982 Method 5 audits, expressed as the percent difference from the true (EPA) value at various levels of accuracy. The Code of Federal Regulations (1) requires that the dry gas meter be calibrated with an accuracy of $\pm$ 2 percent. Figure 1 shows that 44% of the reporting laboratories in the 0382 audit and 43% in the 0982 audit obtained this accuracy. These results are similar to those in previous audits (Figure 2). Ninety-two of the laboratories participated in both audits.

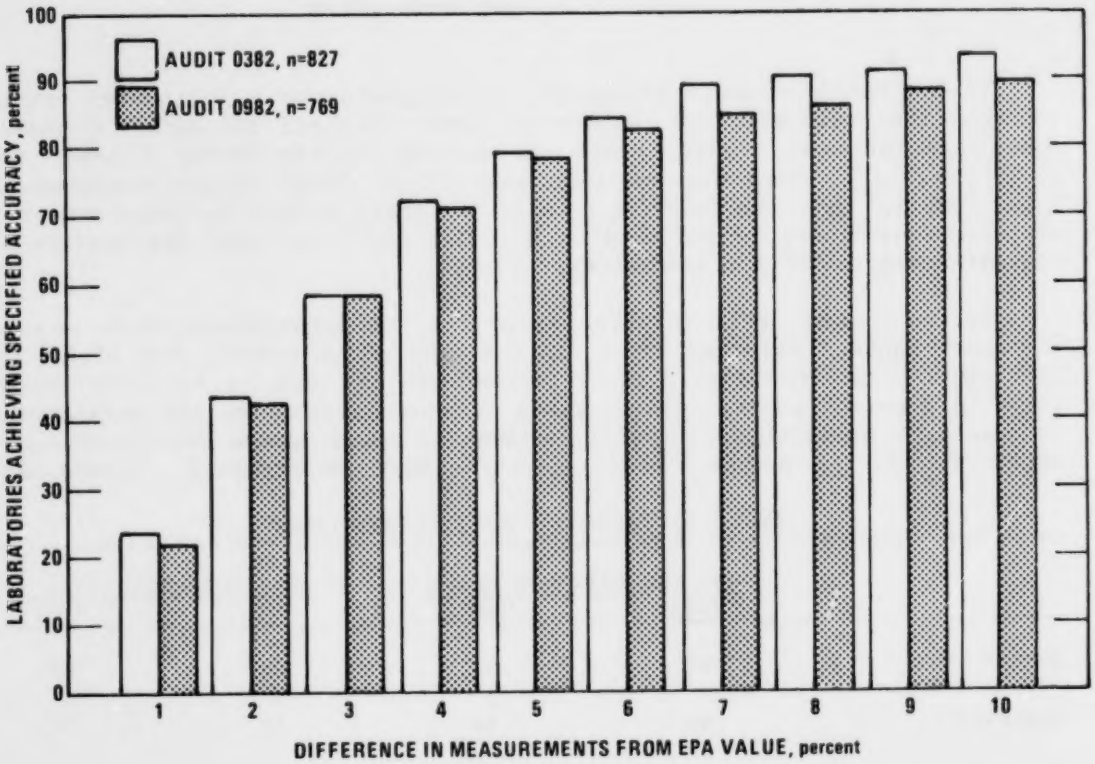

Figure 1. Cumulative accuracy for participants in method 5 audits 0382 and 0982.

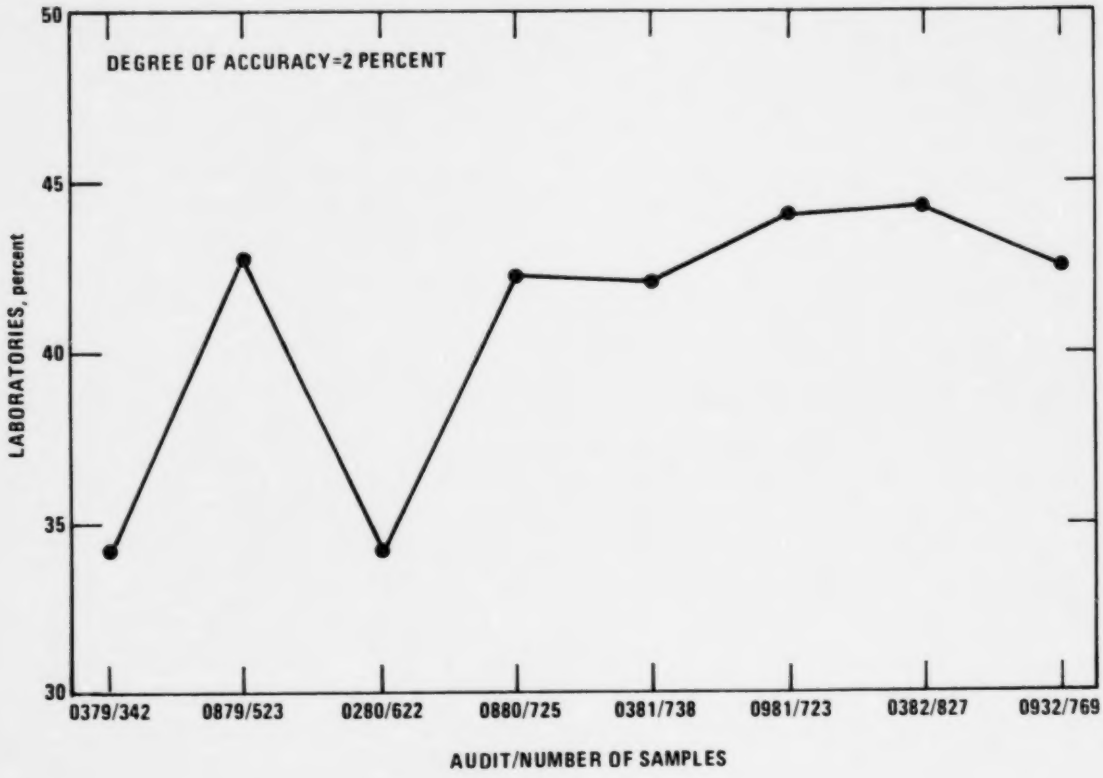

Figure 2. Previous results of method 5 audit.

The histograms in Figures 3 and 4 show how the individual results of the 0382 and 0982 audits compared to the mean and the median values for all participants. The majority of the laboratories reported values higher than the EPA value. The standard deviation of the triplicate analyses (precision) by each laboratory indicated that for the 0382 audit, 70% of the standard deviations for each set were within 0.3%. For the 0982 audit, 74% of the standard deviations were within 0.3%. Six percent of the 0382 data and 8% of the 0982 data were identified as outliers using Chauvenet's Criterion (4). Before the outliers were removed, the mean values for the 0382 and 0982 data differed by 3.5 and 8.5% from the true value, respectively. After deletion of outliers, these values were reduced to 0.2 and 1.3%, respectively.

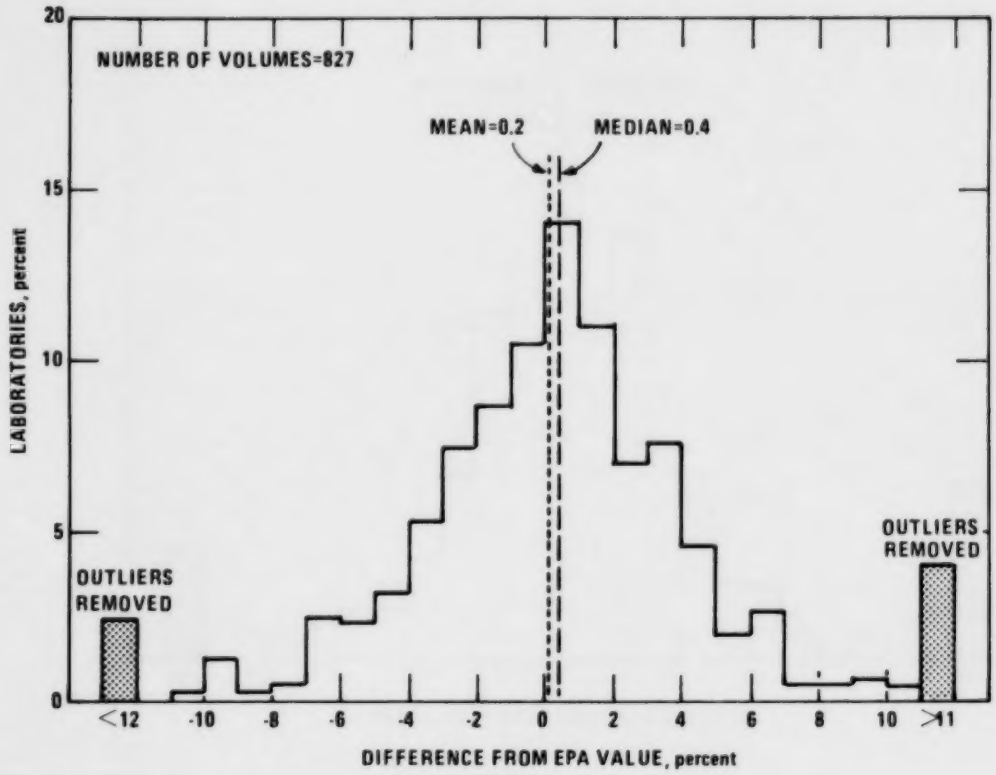

Figure 3. Results of method 5 audit 0382.

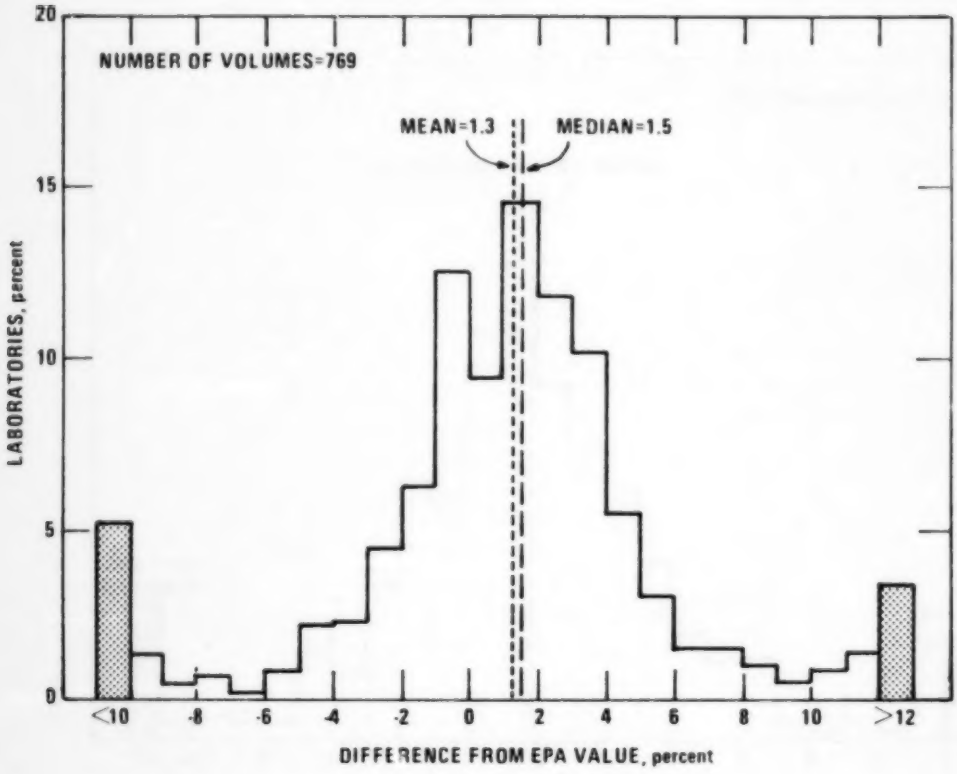

Figure 4. Results of method 5 audit 0982.

SECTION 5

METHOD 6 AUDIT

This audit checks the participant's ability to analyze a Method 6 sample for sulfate. The audit set consists of five dilutions of 10 N sulfuric acid in 25-ml sealed glass ampoules. These five ampoules contain the equivalent of approximately 0 to 3000 mg SO_2/DSCM (dry standard cubic meter). The analyst withdraws 5.0 ml from each sample, adds 30 ml of 3% hydrogen peroxide, and dilutes the sample to 100 ml with distilled water. A 20-ml aliquot is then withdrawn from the diluted sample, 80 ml of 100% isopropanol and thorin indicator are added, and the sample is titrated with barium perchlorate ($Ba[ClO_4]_2$) to a pink end point. In calculating the results, the participants assume they had an original sample volume of 100 ml, and that they sampled 21 x 10^{-3} DSCM (dry standard cubic meter) of stack gas.

In the spring audit (0282), 74% of the 156 laboratories that received the audit package returned data. In the fall audit (0882), 67% of the 156 laboratories returned data. These percentages are similar to those encountered in previous audits (2,3). Table 6, which classifies the participants into general categories, shows the total number of participants requesting participation and the number that returned data. Eighty laboratories participated in both audits and returned data.

TABLE 6. METHOD 6 AUDIT PARTICIPANTS

	No. requesting samples		No. returning data	
	0282	0882	0282	0882
Contractors	90	86	67	48
Industry	42	42	29	29
Foreign	2	4	2	4
Federal	2	2	1	2
State	11	13	10	11
Local	9	9	7	9
TOTAL	156	156	116	103

Table 7 presents the percent of laboratories that achieved 2% and 5% accuracy for each of the five different concentrations in the two 1982 Method 6 audits. The table shows that 68% of the reporting laboratories in the 0282 audit achieved an accuracy within 2% for the higher concentration and that in the 0282 audit, 24% of the laboratories achieved an accuracy within 2%. However, in both audits, approximately 45% of the participants achieved an accuracy within 2% for the lowest concentration samples. Approximately 75% of the laboratories were able to achieve an accuracy level of within 5% on 8 of the 10 samples.

TABLE 7. SUMMARY OF SOURCE SO_2 AUDITS

Concentration	0282		0882	
	±2%	±5%	±2%	±5%
0-500 mg/DSCM	48%	75%	45%	75%
501-1000 mg/DSCM	59%	80%	62%	82%
1001-1500 mg/DSCM	55%	85%	60%	83%
1501-2000 mg/DSCM	68%	88%	20%	51%
2001-3000 mg/DSCM	68%	91%	24%	54%
n	116		103	

The results obtained in the Fall 1982 Method 6 audit did differ from those obtained in previous audits (Figure 5).

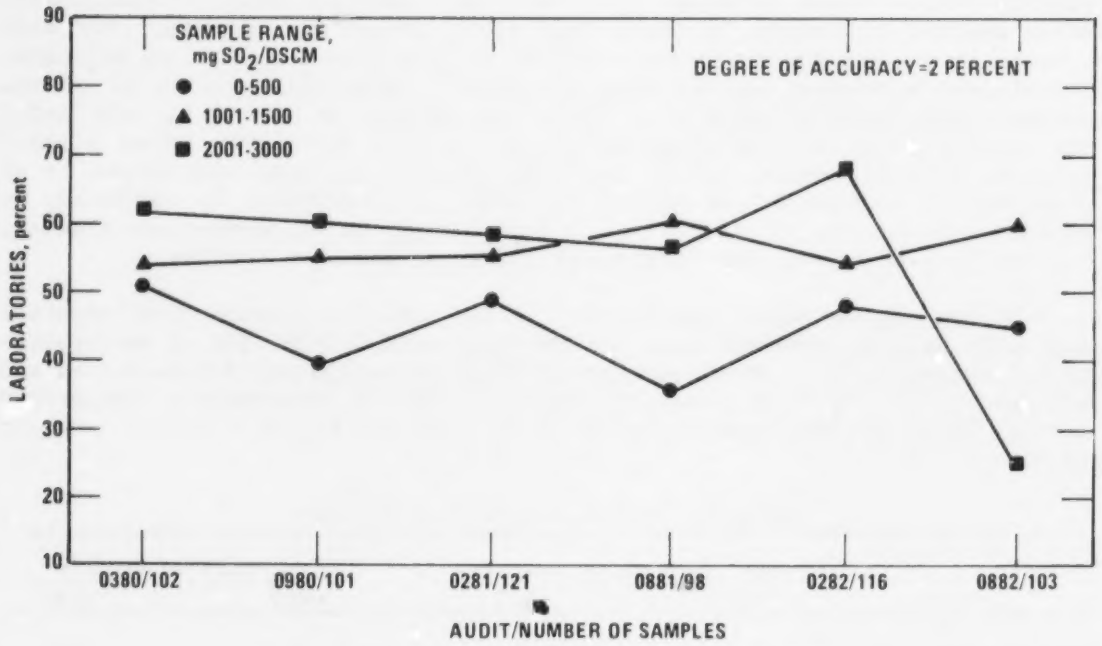

Figure 5. Previous results of method 6 audit.

SECTION 6

METHOD 7 AUDIT

This audit checks the participant's ability to analyze a Method 7 sample for nitrate. The NO_x audit set consists of five dilutions of potassium nitrate (KNO_3) stock solution in 25-ml glass ampoules that are autoclaved after sealing to destroy bacteria that might attack the nitrate. The five samples in the set simulate source samples ranging from 0 to 1000 mg NO_x/DSCM. The analyst withdraws 5.0 ml from an ampoule, adds this and 25 ml of the Method 7 absorbing solution to a flask, adjusts the pH to 9 to 12 with NaOH, and dilutes to 50 ml with distilled water. He then withdraws a 25-ml aliquot from the diluted sample, places it in an evaporating dish, and treats it as described in Section 4.3 of Method 7. After the treatment is completed, he measures the absorbance at 410 nm. In calculating the concentrations present, the participant asumes that 2000 ml of stack gas has been sampled.

In the spring audit (0482), 61% of the 129 laboratories that received the audit package returned data. In the fall audit (1082), 52% of the laboratories returned data. Fifty-one laboratories participated in both audits and returned data. Table 8 shows the total number of laboratories requesting participation and the number that returned data for Method 7 audits 0482 and 1082.

TABLE 8. METHOD 7 AUDIT PARTICIPANTS

	No. requesting samples		No. returning data	
	0482	1082	0482	1082
Contractors	80	76	46	34
Industry	31	30	19	19
Foreign	2	2	1	2
Federal	1	1	1	1
State	8	7	6	4
Local	7	7	6	4
TOTAL	129	123	79	64

Table 9 shows the percent of laboratories that can achieve 3 and 7% accuracy for each of the five concentrations. The table shows that 36% of the reporting laboratories in the 0482 audit achieved an accuracy within 3% on the lowest concentration. In the 1082 audit, 31% of the laboratories achieved an accuracy within 3%. Sixty percent of the laboratories were able to achieve 7% accuracy on all samples in both audits.

TABLE 9. SUMMARY OF SOURCE NO_x AUDITS

Concentration	0482		1082	
	±3%	±7%	±3%	±7%
0-200 mg/DSCM	36%	59%	31%	55%
201-400 mg/DSCM	41%	66%	38%	61%
401-600 mg/DSCM	41%	65%	42%	64%
601-800 mg/DSCM	40%	65%	36%	67%
801-1000 mg/DSCM	36%	65%	37%	61%
n	79		64	

Figure 6 compares the results of the 1982 audit to those of the past four audits (2,3). The results have improved and the percent of laboratories obtaining an accuracy of 3% has held steady, between 30 to 40%.

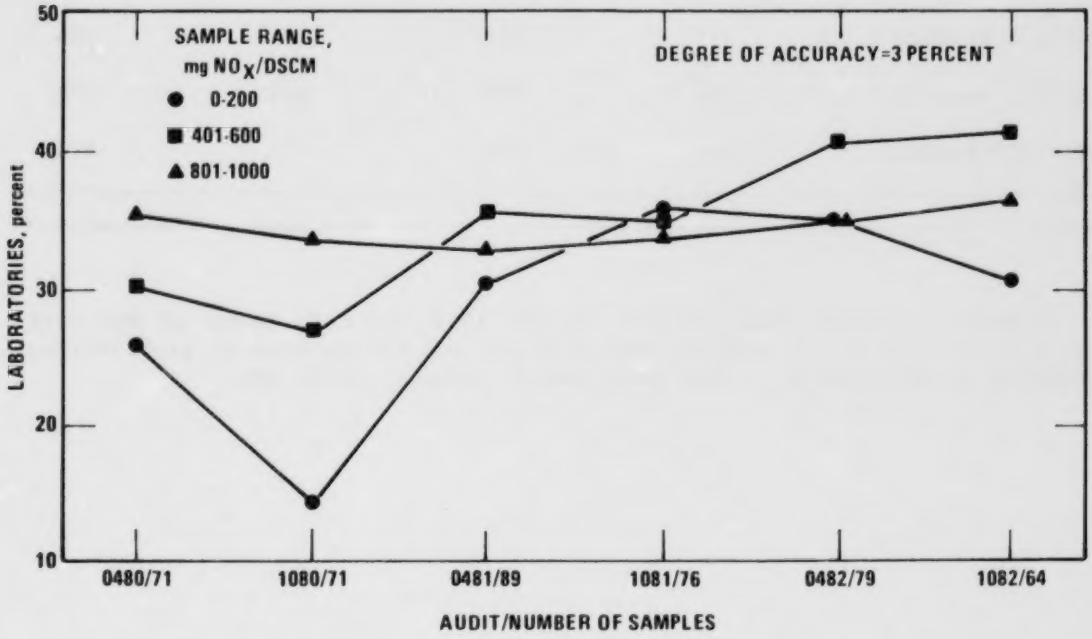

Figure 6. Previous results of method 7 audit.

SECTION 7

METHOD 19A COAL AUDIT

A proposed revision in Part 60, Title 40 of the Code of Federal Regulations will allow coal sampling and analysis to serve as an acceptable method for demonstrating compliance with the SO_2 emissions standard for large Subpart Da coal-fired power plants. The coal audit checks the participant's ability to analyze a coal sample for sulfur, ash, moisture, and BTU content. Acceptance Testing on the commercially obtained coal was done by EPA contractors.

Each set of coal samples consisted of two bottles containing 50 g of 60-mesh coal. Participants measured each sample for sulfur, moisture, ash, and gross calorific content. The following American Society for Testing and Materials (ASTM) procedures were recommended but not necessarily mandated (5).

- ASTM D-3177 (Standard Test Method for Total Sulfur in the Analysis of Coal and Coke);
- ASTM D-3174 (Standard Test Method for Ash in the Analysis Sample of Coal and Coke);
- ASTM D-3173 (Test for Moisture in the Analysis Sample of Coal); and
- ASTM D-2015 (Standard Test Method for Gross Calorific Value of Solid Fuel by the Adiabatic Bomb Method) (5).

The participants measured the four parameters and reported their results for moisture (%) on an as-received basis, and their results for sulfur (%), ash (%), and gross calorific value (BTU/lb) on a dry basis.

In the spring audit (0382), 73% of the 77 laboratories that received the audit package returned data. In the fall audit (0982), 77% of the 90 laboratories returned data. Forty-seven laboratories participated in both audits and returned data. Table 10 shows the total number of laboratories requesting participation and the number that returned data for coal audits 0382 and 0982.

TABLE 10. COAL AUDIT PARTICIPANTS

	No. requesting samples		No. returning data	
	0382	0982	0382	0982
Contractors	33	36	19	22
Industry	30	39	27	36
Federal	2	1	0	0
State	7	10	7	7
Local	5	4	3	4
TOTAL	77	90	56	69

Tables 11 and 12 summarize the coal audit results. The N value is greater than the number of participants because some companies had more than one laboratory participating. In this case, each laboratory received its own set of samples. Each laboratory was asked to analyze the samples in duplicate. Accuracies of 5% and 10% were chosen for the precision criteria for each of the four parameters.

In both audits, 50% of the laboratories were able to analyze the sulfur content of the low level samples within 5% of the expected value. Seventy percent of the laboratories achieved 5% accuracy for the high level sulfur concentrations. Only 10% of the laboratories achieved 5% on both of the moisture concentrations. For the ash analysis and BTU content, 95% to 100% of the reporting laboratories were able to achieve an accuracy within 5% for both sample concentrations.

The participants' accuracy improved with higher concentrations on all four parameters. For those that did duplicate analyses, the intra-laboratory precision showed no correlation with concentration. Therefore, the standard deviation (precision) was independent of the sample concentration for all four parameters.

TABLE 11. SOURCE COAL AUDIT - 0382

Expected value	No. of analyses	Laboratories accurate within ±5%	Laboratories accurate within ±10%
		Sulfur	
1.30%	(1) 73	48%	85%
	(2) 64	44%	84%
2.85%	(1) 73	68%	88%
	(2) 64	69%	86%
		Moisture	
0.96%	(1) 73	10%	23%
	(2) 63	6%	19%
1.73%	(1) 73	22%	42%
	(2) 63	19%	41%
		Ash	
21.48%	(1) 73	97%	100%
	(2) 64	95%	98%
35.40%	(1) 73	99%	100%
	(2) 64	100%	100%
		Gross calorific	
9322 BTU/lb	(1) 71	100%	100%
	(2) 62	100%	100%
11278 BTU/lb	(1) 71	100%	100%
	(2) 62	100%	100%

TABLE 12. SOURCE COAL AUDIT - 0982

Expected value	No. of analyses	Laboratories accurate within ±5%	Laboratories accurate within ±10%
		Sulfur	
1.22%	(1) 81	57%	79%
	(2) 78	53%	76%
3.22%	(1) 81	73%	90%
	(2) 78	78%	92%
		Moisture	
2.11%	(1) 82	17%	32%
	(2) 76	12%	29%
2.48%	(1) 82	17%	40%
	(2) 76	20%	39%
		Ash	
11.43%	(1) 81	98%	100%
	(2) 78	94%	100%
16.34%	(1) 81	99%	99%
	(2) 78	97%	99%
		Gross calorific	
12277 BTU/lb	(1) 78	97%	100%
	(2) 76	96%	100%
12932 BTU/lb	(1) 78	100%	100%
	(2) 76	99%	100%

SECTION 8

METHOD 3 AUDIT

This audit checks the participant's ability to analyze a gas sample using an Orsat analyzer. The audit package consists of a disposable 4-liter cylinder and a Tedlar sample bag. The analyst expels the gas from the cylinder into the sample bag. Then a gas sample is extracted from the bag into the Orsat analyzer. The gas sample is analyzed for percent carbon dioxide, oxygen, and carbon monoxide. This was the first Method 3 audit conducted by QAD.

In this audit, 59% of the 96 laboratories that received the audit package returned data. Table 13 shows the total number of laboratories requesting participation and the number that returned data for the Method 3 audit.

TABLE 13. METHOD 3 AUDIT PARTICIPANTS

	No. requesting samples 0682	No. returning data 0682
Contractors	57	29
Industry	25	17
Foreign	1	1
Federal	2	1
State	8	6
Local	3	3
TOTAL	96	57

Table 14 summarizes the Method 3 audit. Each laboratory was asked to analyze the sample in duplicate. Some laboratories exhausted their gas sample after the first analysis. Five and ten percent accuracy were chosen for the precision criteria for each of the three parameters. Each parameter had only one concentration.

Sixty-five percent of the reporting laboratories were able to analyze the percent CO_2 within an accuracy of 10%. For the percent O_2 analysis, only 45% of the laboratories achieved an accuracy within 10%. Since CO was analyzed last and the concentration was higher than for normal conditions, only 31% of the laboratories achieved an accuracy within 10%, and 15% of the participants did not report a value for CO.

TABLE 14. SOURCE METHOD 3 AUDIT - 0682

Expected value	No. of analyses	Laboratories accurate within ±5%	Laboratories accurate within ±10%
		CO_2	
14.8%	(1) 59	34%	68%
	(2) 56	34%	66%
		O_2	
5.2%	(1) 57	28%	49%
	(2) 54	28%	43%
		CO	
7.0%	(1) 50	14%	30%
	(2) 47	13%	32%

REFERENCES

1. U.S. Environmental Protection Agency. Standards of Performance for New Stationary Sources - Appendix A. Title 40, Part 60, Code of Federal Regulations.

2. Fuerst, R. G., E. W. Streib, and M. R. Midgett. A Summary of the EPA National Source Performance Audit Program - 1979. EPA-600/4-81-029, U.S. Environmental Protection Agency, Research Triangle Park, NC 27711, April 1981. 53 pp.

3. Fuerst, R. G. E. W. Streib, and M. R. Midgett. A Summary of the EPA National Source Performance Audit Program - 1980. EPA-600/4-81-077, U.S. Environmental Protection Agency, Research Triangle Park, NC 27711, December 1981.

4. Chauvenet, W. Manual of Spherical and Practical Astronomy: Volume II - Theory and Use of Astronomical Instruments (Method of Least Squares). J. B. Lippincott and Co., Philadelphia, PA 1863(?). pp. 558-565.

5. American Society for Testing and Materials. Annual Book of ASTM Standards - 1979. Part 26. 01-026079-13, American Society for Testing and Materials, Philadelphia, PA.

APPENDIX A

FREQUENCY DISTRIBUTIONS

DGM FREQUENCY DISTRIBUTION OF ABSOLUTE PERCENT DIFFERENCE - 0382

#SAMP	MIN	10%	20%	30%	40%	50%	60%	70%	80%	90%	MAX	MEAN	STD. DEV.	SKEWNESS	MEDIAN
827	.0	.4	.8	1.2	1.8	2.5	3.1	3.9	5.1	7.6	596.0	7.6	39.5	10.82	2.5
820	.0	.4	.8	1.2	1.8	2.4	3.1	3.8	5.0	7.1	51.6	4.1	6.6	-.00	2.4
803	.0	.4	.8	1.2	1.8	2.4	3.0	3.7	4.7	6.5	25.3	3.3	3.7	-.02	2.4
786	.0	.4	.8	1.2	1.7	2.3	3.0	3.6	4.5	6.3	15.2	2.9	2.6	-.03	2.3
775	.0	.4	.8	1.2	1.7	2.3	3.0	3.5	4.3	6.1	11.1	2.8	2.3	-.04	2.3
771	.0	.4	.8	1.2	1.6	2.3	2.9	3.5	4.3	6.0	10.2	2.7	2.2	-.05	2.3
769	.0	.4	.8	1.2	1.6	2.2	2.9	3.5	4.3	6.0	10.0	2.7	2.2	-.06	2.3
766	.0	.4	.8	1.2	1.6	2.2	2.9	3.4	4.2	5.9	9.8	2.7	2.1	-.08	2.2
764	.0	.4	.8	1.2	1.6	2.2	2.9	3.4	4.2	5.8	9.7	2.7	2.1	-.10	2.2
762	.0	.4	.8	1.2	1.6	2.2	2.9	3.4	4.2	5.7	9.6	2.6	2.1	-.11	2.2
759	.0	.4	.8	1.2	1.6	2.2	2.8	3.4	4.2	5.6	9.4	2.6	2.0	-.13	2.2
755	.0	.4	.8	1.1	1.6	2.2	2.8	3.4	4.1	5.5	9.3	2.6	2.0	-.15	2.2
752	.0	.4	.8	1.1	1.6	2.2	2.8	3.4	4.1	5.5	9.0	2.6	1.9	-.17	2.2
751	.0	.4	.8	1.1	1.6	2.2	2.8	3.4	4.1	5.5	8.9	2.6	1.9	-.19	2.2

DGM FREQUENCY DISTRIBUTION OF ABSOLUTE PERCENT DIFFERENCE - 0982

#SAMP	MIN	10%	20%	30%	40%	50%	60%	70%	80%	90%	MAX	MEAN	STD. DEV.	SKEWNESS	MEDIAN
769	.0	.4	1.0	1.5	1.9	2.5	3.2	4.0	5.4	10.5	933.8	12.5	81.4	10.81	2.5
763	.0	.4	.9	1.5	1.9	2.5	3.1	4.0	5.3	10.0	96.4	5.3	12.3	.00	2.5
748	.0	.4	.9	1.4	1.9	2.4	3.0	3.8	4.9	8.9	29.1	3.7	4.4	-.01	2.4
728	.0	.4	.9	1.4	1.8	2.3	2.9	3.6	4.5	7.3	17.8	3.2	3.2	-.01	2.3
715	.0	.4	.9	1.4	1.8	2.3	2.8	3.6	4.3	6.9	12.9	3.0	2.7	-.01	2.3
710	.0	.4	.9	1.4	1.8	2.3	2.8	3.5	4.3	6.8	11.9	2.9	2.6	-.02	2.3
702	.0	.4	.9	1.3	1.8	2.2	2.8	3.5	4.2	5.8	11.4	2.8	2.4	-.02	2.2
694	.0	.4	.8	1.3	1.7	2.2	2.8	3.4	4.1	5.6	10.6	2.7	2.3	-.03	2.2
688	.0	.4	.8	1.3	1.7	2.2	2.7	3.4	4.1	5.5	10.1	2.7	2.2	-.03	2.2
682	.0	.4	.8	1.3	1.7	2.2	2.7	3.3	4.0	5.4	9.8	2.6	2.1	-.04	2.2
680	.0	.4	.8	1.3	1.7	2.2	2.7	3.3	4.0	5.4	9.4	2.6	2.0	-.04	2.2
677	.0	.4	.8	1.3	1.7	2.1	2.7	3.3	4.0	5.3	9.1	2.6	2.0	-.05	2.2
676	.0	.4	.8	1.3	1.7	2.1	2.7	3.3	4.0	5.2	9.0	2.6	2.0	-.06	2.1
675	.0	.4	.8	1.3	1.7	2.1	2.7	3.3	4.0	5.2	9.0	2.5	1.9	-.06	2.1
674	.0	.4	.8	1.3	1.7	2.1	2.7	3.3	4.0	5.1	8.9	2.5	1.9	-.07	2.1
672	.0	.4	.8	1.3	1.7	2.1	2.7	3.3	4.0	5.1	8.9	2.5	1.9	-.08	2.1
670	.0	.4	.8	1.3	1.7	2.1	2.7	3.2	4.0	5.0	8.7	2.5	1.9	-.09	2.1
669	.0	.4	.8	1.3	1.7	2.1	2.6	3.2	4.0	5.0	8.7	2.5	1.9	-.09	2.1
667	.0	.4	.8	1.3	1.7	2.1	2.6	3.2	4.0	5.0	8.5	2.5	1.8	-.10	2.1
666	.0	.4	.8	1.3	1.7	2.1	2.6	3.2	3.9	4.9	8.5	2.5	1.8	-.11	2.1
665	.0	.4	.8	1.3	1.7	2.1	2.6	3.2	3.9	4.9	8.3	2.5	1.8	-.12	2.1

SO_2 FREQUENCY DISTRIBUTION OF PERCENT DIFFERENCE WITH OUTLIERS REMOVED

AUDIT 0282

LEVEL (mg/DSCM)	NOBS	MIN	10%	20%	30%	40%	50%	60%	70%	80%	90%	MAX	AVE	STD
.1- 500.0	103	.00	.16	.60	1.00	1.49	1.91	2.54	3.33	4.59	6.43	9.89	2.73	2.48
500.1-1000.0	89	.01	.22	.57	.74	1.08	1.32	1.53	1.74	2.18	2.78	3.92	1.45	.96
1000.0-1500.0	105	.01	.30	.73	1.03	1.30	1.47	1.93	2.22	2.89	4.12	6.00	1.95	1.46
1500.1-2000.0	101	.00	.16	.29	.40	.81	1.08	1.32	1.59	2.03	2.81	4.41	1.30	1.11
2000.1-3000.0	105	.03	.18	.35	.62	.74	1.11	1.46	1.80	2.45	3.49	4.93	1.48	1.25

SO_2 FREQUENCY DISTRIBUTION OF PERCENT DIFFERENCE WITH OUTLIERS REMOVED

AUDIT 0882

LEVEL (mg/DSCM)	NOBS	MIN	10%	20%	30%	40%	50%	60%	70%	80%	90%	MAX	AVE	STD
.1- 500.0	87	.00	.11	.45	1.12	1.42	1.83	2.46	2.80	3.54	6.19	7.87	2.40	2.03
500.1-1000.0	83	.02	.23	.46	.72	.96	1.23	1.45	1.68	2.05	2.85	4.03	1.41	1.01
1000.1-1500.0	81	.07	.25	.42	.59	.85	1.06	1.36	1.68	2.18	2.63	3.16	1.29	.89
1500.1-2000.0	100	.06	1.15	1.84	2.92	3.45	4.55	5.95	7.30	8.81	11.49	14.94	5.58	3.91
2000.1-3000.0	101	.59	1.17	1.58	2.20	2.77	3.64	6.12	7.04	8.13	9.26	11.96	4.85	3.22

NO_x FREQUENCY DISTRIBUTION OF PERCENT DIFFERENCE WITH OUTLIERS REMOVED

AUDIT 0482

LEVEL (mg/DSCM)	NOBS	MIN	10%	20%	30%	40%	50%	60%	70%	80%	90%	MAX	AVE	STD
.1- 200.0	61	.11	.45	1.12	1.79	2.62	3.40	4.24	6.25	7.53	11.33	16.07	4.84	4.23
200.1- 400.0	62	.06	.36	.89	1.73	2.20	2.82	3.82	4.61	5.44	8.18	10.52	3.70	2.89
400.1- 600.0	61	.04	.45	1.25	1.99	2.29	2.51	3.48	4.37	6.57	8.67	12.09	3.87	3.13
600.1- 800.0	66	.01	.62	1.38	2.05	2.77	3.11	4.19	5.47	7.56	11.19	15.15	4.77	4.02
800.1-1000.0	68	.09	.24	.87	1.63	2.82	3.64	4.14	5.28	7.88	12.02	16.86	4.95	4.57

NO_x FREQUENCY DISTRIBUTION OF PERCENT DIFFERENCE WITH OUTLIERS REMOVED

AUDIT 1082

LEVEL (mg/DSCM)	NOBS	MIN	10%	20%	30%	40%	50%	60%	70%	80%	90%	MAX	AVE	STD
.1- 200.0	54	.29	1.00	1.36	2.44	3.16	3.80	5.45	7.68	11.91	17.65	20.52	6.82	6.30
200.1- 400.0	51	.03	.44	1.22	1.73	2.17	2.98	3.92	5.87	7.06	9.89	13.03	4.31	3.51
400.1- 600.0	45	.15	.27	.63	1.07	1.80	2.05	2.97	3.52	5.06	6.95	9.29	3.00	2.53
600.1- 800.0	45	.07	.20	.79	1.44	2.22	2.71	4.16	4.44	5.57	6.15	9.29	3.33	2.39
800.1-1000.0	52	.12	.43	.98	1.54	2.11	3.35	5.06	5.95	7.62	12.17	17.99	5.27	4.97

NATIONAL COAL AUDIT FREQUENCY DISTRIBUTION OF ABSOLUTE PERCENT DIFFERENCES OF EXPECTED AND REPORTED VALUES

AUDIT 0382

	NO.	MIN	10%	20%	30%	40%	50%	60%	70%	80%	90%	MAX	MEAN	STD DEV
Sulfur														
Sample 3000	137	.00	.35	1.05	1.75	2.11	2.81	3.86	5.26	7.02	12.28	96.84	5.97	12.22
Sample 8000	137	.00	.77	3.08	3.85	4.62	5.38	6.15	7.69	9.23	12.31	93.08	7.77	11.59
Moisture														
Sample 3000	136	1.04	5.21	9.38	14.58	18.75	20.83	23.96	28.13	34.38	50.00	72.92	24.34	16.99
Sample 8000	136	1.16	3.47	4.62	6.94	9.25	12.72	14.45	16.76	20.23	32.95	61.27	15.29	12.87
Ash														
Sample 3000	137	.00	.08	.17	.28	.34	.51	.62	.79	.99	1.53	8.11	.72	.89
Sample 8000	137	.00	.23	.47	.79	1.02	1.40	1.68	2.00	2.33	2.75	13.83	1.64	1.60
Gross Calorific														
Sample 3000	133	.01	.39	.68	.89	.99	1.08	1.15	1.27	1.45	1.74	3.14	1.11	.55
Sample 8000	133	.01	.43	.71	.99	1.15	1.29	1.51	1.66	1.91	2.17	2.66	1.32	.63

NATIONAL COAL AUDIT FREQUENCY DISTRIBUTION OF ABSOLUTE PERCENT DIFFERENCES OF EXPECTED AND REPORTED VALUES

AUDIT 0982

	NO.	MIN	10%	20%	30%	40%	50%	60%	70%	80%	90%	MAX	MEAN	STD DEV
Sulfur														
Sample 4000	159	.00	.31	.62	1.24	1.86	2.48	3.11	4.66	6.21	9.01	39.75	4.14	5.45
Sample 5000	159	.00	.82	1.64	2.46	3.28	4.92	6.56	8.20	11.48	16.39	161.48	10.39	25.47
Moisture														
Sample 4000	158	.47	3.32	6.16	9.95	13.74	16.11	18.48	21.80	24.64	32.23	141.23	19.29	19.05
Sample 5000	158	.00	3.23	5.24	7.66	9.68	12.90	15.73	18.55	21.37	26.21	116.94	15.58	15.64
Ash														
Sample 4000	159	.17	.52	.79	1.05	1.40	1.66	1.92	2.36	2.80	3.50	15.40	2.00	1.72
Sample 5000	159	.00	.18	.37	.61	.98	1.16	1.47	1.65	2.20	2.94	31.33	1.74	3.49
Gross Calorific														
Sample 4000	154	.00	.06	.12	.21	.29	.42	.60	.94	1.58	2.34	5.22	.85	.98
Sample 5000	154	.01	.09	.18	.27	.37	.48	.60	.85	1.60	2.36	5.78	.95	1.23

NATIONAL ORSAT AUDIT FREQUENCY DISTRIBUTION OF ABSOLUTE PERCENT DIFFERENCES OF EXPECTED AND REPORTED VALUES

AUDIT 0682

	NO.	MIN	10%	20%	30%	40%	50%	60%	70%	80%	90%	MAX	MEAN	STD DEV
							CO_2							
Sample 6000	115	.00	1.35	2.70	4.05	5.41	6.76	8.11	10.14	12.16	23.65	58.78	10.44	12.10
							O_2							
Sample 6000	111	.00	1.92	3.85	5.77	7.69	11.54	15.38	19.23	26.92	53.85	134.62	20.06	25.22
							CO							
Sample 6000	97	.00	2.86	8.57	10.00	12.86	18.57	22.86	30.00	57.14	74.29	97.14	29.47	27.37

APPENDIX B

NATIONAL ORSAT (METHOD 3) AUDIT STATISTICS

STUDY ID: 0682

Parameter: CO_2	Mean: 13.25	Coef. Var.: 13.81
Sample Number: 6000	Median: 13.90	Upper Conf. Int.: 13.72
Analysis: 1	Range: 8.50	Lower Conf. Int.: 12.79
Number of OBS: 59	Variance: 3.35	Skewness: -2.28
Expected Value: 14.80	Std. Dev.: 1.83	Accuracy: -6.08

---------- DATA IN ASCENDING ORDER ----------

6.30	7.20	7.60	8.80	10.80
11.20	11.30	11.60	12.00	12.00
12.60	13.00	13.00	13.20	13.20
13.20	13.30	13.30	13.30	13.40
13.40	13.50	13.50	13.60	13.60
13.70	13.70	13.80	13.80	13.90
13.90	13.90	13.90	14.00	14.00
14.00	14.00	14.00	14.00	14.20
14.20	14.20	14.20	14.30	14.30
14.40	14.40	14.40	14.40	14.40
14.50	14.50	14.60	14.60	14.70
14.70	14.80	14.80	14.80	

STUDY ID: 0682

Parameter: CO_2	Mean: 13.29	Coef. Var.: 13.52
Sample Number: 6000	Median: 13.75	Upper Conf. Int.: 13.77
Analysis: 2	Range: 9.70	Lower Conf. Int.: 12.82
Number of OBS: 56	Variance: 3.23	Skewness: -2.19
Expected Value: 14.80	Std. Dev.: 1.80	Accuracy: -7.09

---------- DATA IN ASCENDING ORDER ----------

6.10	7.20	9.20	9.50	10.60
11.00	11.40	12.00	12.50	12.50
12.80	12.80	13.00	13.20	13.20
13.20	13.20	13.20	13.30	13.40
13.40	13.40	13.60	13.60	13.60
13.70	13.70	13.70	13.80	13.80
14.00	14.00	14.00	14.00	14.00
14.00	14.10	14.20	14.20	14.30
14.30	14.40	14.40	14.40	14.40
14.50	14.50	14.50	14.50	14.60
14.70	14.70	14.80	14.80	14.80
15.80				

STUDY ID: 0682

Parameter: O_2	Mean: 6.11	Coef. Var.: 22.17
Sample Number: 6000	Median: 5.70	Upper Conf. Int.: 6.46
Analysis: 1	Range: 7.80	Lower Conf. Int.: 5.76
Number of OBS: 57	Variance: 1.83	Skewness: 2.41
Expected Value: 5.20	Std. Dev.: 1.35	Accuracy: 9.62

DATA IN ASCENDING ORDER

4.40	4.60	4.80	5.00	5.00
5.20	5.20	5.20	5.30	5.30
5.30	5.30	5.30	5.30	5.30
5.40	5.40	5.40	5.40	5.50
5.50	5.50	5.50	5.60	5.60
5.60	5.60	5.70	5.70	5.70
5.80	5.90	5.90	6.00	6.00
6.00	6.00	6.00	6.10	6.20
6.20	6.20	6.30	6.40	6.40
6.50	6.60	6.60	6.80	7.00
7.60	8.00	8.00	8.40	8.90
10.50	12.20			

STUDY ID: 0682

Parameter: O_2	Mean: 6.20	Coef. Var.: 22.90
Sample Number: 6000	Median: 5.80	Upper Conf. Int.: 6.58
Analysis: 2	Range: 6.80	Lower Conf. Int.: 5.82
Number of OBS: 54	Variance: 2.01	Skewness: 1.70
Expected Value: 5.20	Std. Dev.: 1.42	Accuracy: 11.54

DATA IN ASCENDING ORDER

4.30	4.30	4.60	5.00	5.00
5.10	5.20	5.20	5.20	5.30
5.30	5.30	5.30	5.30	5.40
5.40	5.40	5.40	5.50	5.50
5.60	5.60	5.60	5.60	5.70
5.70	5.80	5.80	5.80	5.90
6.00	6.00	6.10	6.10	6.20
6.20	6.20	6.30	6.30	6.50
6.50	6.60	6.60	6.60	6.60
7.00	7.40	8.00	8.40	8.80
9.00	9.60	10.50	11.10	

STUDY ID: 0682

Parameter: CO	Mean: 5.06	Coef. Var.: 38.82
Sample Number: 6000	Median: 5.65	Upper Conf. Int.: 5.60
Analysis: 1	Range: 7.80	Lower Conf. Int.: 4.52
Number of OBS: 50	Variance: 3.86	Skewness: -.91
Expected Value: 7.00	Std. Dev.: 1.96	Accuracy: -19.29

---------- DATA IN ASCENDING ORDER ----------

.20	.20	1.20	1.30	1.80
2.00	2.50	2.80	2.80	3.00
3.10	3.40	4.10	4.10	4.60
4.90	5.00	5.00	5.00	5.20
5.40	5.40	5.40	5.50	5.60
5.70	5.80	6.00	6.10	6.10
6.20	6.20	6.20	6.30	6.30
6.40	6.40	6.40	6.40	6.50
6.60	6.70	6.80	6.80	6.80
6.80	6.90	7.10	8.00	8.00

STUDY ID: 0682

Parameter: CO	Mean: 4.96	Coef. Var.: 41.27
Sample Number: 6000	Median: 5.70	Upper Conf. Int.: 5.55
Analysis: 2	Range: 7.50	Lower Conf. Int.: 4.38
Number of OBS: 47	Variance: 4.20	Skewness: -.86
Expected Value: 7.00	Std. Dev.: 2.05	Accuracy: -18.57

---------- DATA IN ASCENDING ORDER ----------

.20	.30	.90	1.30	1.50
1.60	2.20	2.80	3.00	3.00
3.00	3.40	3.60	3.90	4.00
5.00	5.00	5.10	5.20	5.40
5.50	5.60	5.60	5.70	5.80
5.80	6.00	6.10	6.20	6.20
6.20	6.20	6.30	6.30	6.40
6.40	6.40	6.60	6.70	6.80
6.80	6.80	6.90	7.00	7.40
7.50	7.70			

APPENDIX C

INSTRUCTIONS FOR EPA AUDIT MATERIALS

INSTRUCTIONS FOR USE OF ENVIRONMENTAL PROTECTION AGENCY METHOD 5 DRY GAS METER PERFORMANCE TEST DEVICE

NOTE: All procedures referrred to are from revised Method 5 published in the Federal Register, Vol. 12, No. 160, Part II, Thursday, August 18, 1977, pp. 41776-41782 and references contained therein. This revised method should be adhered to in all details in the use of this quality assurance performance device.

EQUIPMENT: The participant in this study should possess the following equipment, including the performance test device supplied by EPA.

Quantity	Item
1	Method 5/Source Sampling Meter Box
1	Stopwatch, preferably calibrated in decimal minutes
1	Thermometer, ambient range
1	Barometer. If unavailable, call nearest National Weather Service and request the ABSOLUTE barometric pressure. (Corrected for temperature and acceleration due to gravity, but not corrected for altitude.)
1	Performance Test Device. A calibrated flow orifice housed in a quick-connect coupling and identified with an engraved three-digit serial number. WARNING: THE DEVICE MUST NOT BE DISASSEMBLED UNDER ANY CIRCUMSTANCES. Use these devices at room temperature.

PROCEDURE:

1. Remove the performance test device from its case and insert it into the gas inlet quick-connect coupling on the source sampling meter box.
2. Turn the power to the meter box on and start the pump.
3. Adjust the coarse flow rate control valve and the fine flow rate control valve to give a maximum vacuum reading. CAUTION: A vacuum reading of less than 17 inches Hg will result in flow rate errors.
4. Allow the orifice and source sampling meter box to warm up for 45 minutes with flow controls adjusted as described in Step 3 before starting quality assurance runs.
5. Make triplicate quality assurance runs. For each run, record initial and final dry gas meter volumes, dry gas meter inlet and outlet temperatures, internal orifice pressure drop (ΔH), ambient temperature, and barometric pressure. Run duration should be slightly greater than 15 minutes. The following procedure is recommended. Fifteen minutes after a run is started, the participant watches the dry gas meter needle closely. As the needle reaches the zero (12 o'clock) position, the pump and stopwatch are stopped simultaneously. The dry gas meter volume and time are recorded.
   This complete run procedure is performed three times to provide the required triplicate quality assurance runs.
6. Calculate the corrected dry gas volume for each run using equation 5.1 of the above-referenced Method 5. For each replicate, record the corrected dry gas volume in dry standard cubic meters, the sampling time in decimal minutes, the barometric pressure in mm Hg, and the ambient temperature in degrees Celcius on the enclosed data card. Be sure to record the performance test device serial number on the data card in the column headed "Orifice Number."
   NOTE 1: If you calculate dry gas volume in English Units, use the conversion factor of 0.02832 m^3 ft^3 to obtain the volume in metric units.
   NOTE 2: If your stopwatch is not in decimal minutes, be sure to convert (e.g. 15 minutes 20 seconds is reported as 15.33 minutes).
7. After recording the requested data on the enclosed data form, return the data form and the performance test device to:

   Quality Assurance Division (MD-77)
   Environmental Monitoring Systems Laboratory
   Environmental Protection Agency
   Research Triangle Park, North Carolina 27711
   Attention: Ms. Ellen Streib

   A postpaid return envelope and label are enclosed for this purpose.

INSTRUCTIONS FOR USE OF ENVIRONMENTAL PROTECTION AGENCY STATIONARY SOURCE QUALITY ASSURANCE SO_2 REFERENCE SAMPLES

Note: All Method 6 procedures referred to are from the amended method published in the *Federal Register* Vol. 42, No. 160, Part II, Thursday, August 18, 1977, pp 41782-41784. This amended method should be adhered to in all details in the analysis of these reference standards.

1. Prepare 3-percent hydrogen peroxide according to Section 3.1.3 of the method (30 ml is required for each sample and each blank).

2. Prepare each reference sample for analysis as follows: Wrap a paper towel around the ampule and with the ampule in an upright position break off the top at the prescored mark by exerting pressure sideways. From the ampule pipette exactly 5 ml of the reference sample into a 100-ml volumetric flask. Add 30 ml of 3-percent hydrogen peroxide solution. Dilute exactly to the mark with deionized, distilled water. Analyze the sample in accordance with the procedure detailed in Section 4.3 of the method, beginning with "Pipette a 20-ml aliquot of this solution. . . ." (Note: If more than 50 ml of barium perchlorate titrant is required for any sample analysis, a smaller aliquot should be selected to allow titration with less than 50-ml titrant.)

3. Calculate the concentration, C_{SO_2} (concentration of sulfur dioxide, dry basis, corrected to standard conditions, mg/dscm), using Equation 6-2. A value of 21 X 10^{-3} dscm should be used for $V_{m(std)}$, in the equation. A value of 100 ml should be used for V_{soln} in the equation.

4. Record the reference standard sample numbers and their corresponding SO_2 concentrations in mg/dscm on the enclosed data form. Return the form to:

Quality Assurance Division (MD 77)
Environmental Monitoring Systems Laboratory
Environmental Protection Agency
Research Triangle Park, N.C. 27711
ATTN: Ellen W. Streib

If other than EPA Method 6 is used for your analyses, please explain in detail your analytical procedure on the back of the enclosed data form.

INSTRUCTIONS FOR USE OF ENVIRONMENTAL PROTECTION AGENCY STATIONARY SOURCE QUALITY ASSURANCE NO_x REFERENCE SAMPLES

Note: All Method 7 procedures referred to are from the amended method published in the *Federal Register* Vol. 42, No. 160, Part II, Thursday, August 18, 1977, pp 41784-41786. This amended method should be adhered to in all details in the analysis of these reference standards.

1. Prepare absorbing solution according to Section 3.1 of the method.

2. Prepare each reference sample for analysis as follows: Wrap a paper towel around the ampule and with the ampule in an upright position break off the top at the prescored mark by exerting pressure sideways. From the ampule pipette exactly 5 ml of the reference sample into a 100-ml beaker. Add 25 ml absorbing solution to the beaker; adjust the pH to 9-12 (using pH paper as indicated in Section 4.2 of the method) by dropwise addition of sodium hydroxide (1N). Quantitatively transfer the contents of the beaker to a 50-ml volumetric flask and dilute exactly to the mark with deionized, distilled water. Mix thoroughly and pipette a 25-ml aliquot of the diluted sample into a porcelain evaporating dish. Beginning with the evaporation step in Section 4.3, complete the sample analysis.

3. Calculate total μg NO_2 per sample using Equation 7-3. Calculate the sample concentration, C (concentration of NO_x as NO_2, dry basis, corrected to standard conditions, mg/dscm), using Equation 7-4. A value of 2000 ml should be used for V_{sc} in Equation 7-4.

4. Record the reference sample numbers and their corresponding concentrations, C, in mg/dscm on the enclosed data form. Return the form to:

Quality Assurance Division (MD 77)
Environmental Monitoring Systems Laboratory
Environmental Protection Agency
Research Triangle Park, N.C. 27711
ATTN: Ellen W. Streib

If other than EPA Method 7 is used for your analyses, please explain in detail your analytical procedure on the back of the enclosed data form.

COAL AUDIT PROGRAM INFORMATION

1. There is approximately 50 grams of 60 mesh coal per bottle.

2. Analyze the coal samples for moisture and on a dry basis for ash, sulfur and gross calorific value. Report moisture, ash, and sulfur in weight percent with gross calorific value reported as BTU/lb.

3. All methods used in the analysis of these coal samples should follow American Society for Testing and Materials (ASTM) recommended procedures or an accepted automatic analytical device.

4. Suggested procedures are:

Moisture	D-3173
Ash	D-3174
Sulfur	D-3177
Gross Calorific Value ...	D-2015

   Please note on the data card (columns 17-32) the ASTM method number. If an ASTM method was not used for analysis note that on the back of the data card. Be parameter specific.

5. If you cannot analyze the coal sample for all four parameters, analyze for what you can. Analysis of moisture is necessary to calculate on a dry basis any of the other three parameters. Analysis of sulfur is also necessary for the calculation of gross calorific value.

6. Analyze each sample in duplicate (if possible) and record results as analysis 1 and analysis 2 for each parameter.

7. Most laboratories will use site number 001. Multiple site numbers are used by laboratories that receive more than one set of samples. These central laboratories have requested auditing of their satellite laboratories.

8. After recording the requested data on the enclosed data card, return the data card to:

   Ms. Ellen W. Streib
   Quality Assurance Division (MD-77)
   Environmental Monitoring Systems Laboratory
   U.S. Environmental Protection Agency
   Research Triangle Park, NC 27711

   A postpaid return envelope is enclosed for this purpose.

9. If you have any questions concerning this or any source method audit, please call (919/541-7834).

INSTRUCTIONS FOR USING EPA METHOD 3 AUDIT MATERIALS

Equipment Supplied with Audit Kit

(1) Small gas cylinder containing four liters of gas

(2) Small needle with rubber septum (inside gas cylinder cap)

(3) Tedlar bag, 0.5 ℓ with valve and Tygon tubing

Equipment to be Supplied by Participant

(1) Orsat analyzer

(2) Vacuum source vented to hood for evacuating bag

(3) Container filled with water for checking Tedlar bag for leaks

Procedure

CAUTION: Before performing Step 1 below, become familiar with the operation and performance of the valve on the Tedlar bag. Turning the valve stem clockwise closes the valve. Turning it counterclockwise opens it. This type valve leaks at the stem base when NOT fully closed. Thus, when conducting the leak-check and when the bag is not being filled or emptied, the VALVE MUST BE FULLY CLOSED. Further, when gas is withdrawn from the bag, the bag wall opposite the valve must not block the valve opening because air will then leak into the gas stream at the valve stem base.

(1) Fully open the valve and gently blow into the bag until it is fully inflated. Do not overpressurize the bag!

(2) Close the valve fully.

(3) Immerse the bag in water and determine if it is leaking (as shown by the presence of air bubbles). If the bag, itself, is leaking the leak may be repairable by placing transparent tape over it. If the leak cannot be repaired, do not proceed further. Contact Ms. Ellen Streib at 919/541-7834 for instructions.

(4) After the bag passes the leak-check, evacuate it completely and close the valve. (CAUTION: Gas mixture is toxic (carbon monoxide), do not evacuate by mouth.)

(5) Take the cap from the gas cylinder and remove the plastic tube inside. Place the plastic tube into hole in push button of can with the septum end facing outward.

(6) Insert the septum into the Tygon tubing on the bag valve, open the bag valve and carefully fill the bag with cylinder gas. Do not overpressurize! Fully close the valve and remove the septum/can from the Tygon tubing. Fully evacuate the bag.

(7) Refill the bag with cylinder gas. Fully close the valve and remove the septum/can assembly from the Tygon tubing.

(8) Attach the bag to the Orsat analyzer. Open the bag valve fully and draw 100 cc of gas into the analyzer. Vent the Orsat gas sample to the atmosphere and then refill the Orsat with gas from the Tedlar bag.

(9) Analyze for CO_2, O_2 and CO as described in Sections 4.2.5, 4.2.6 and 4.2.7 of EPA Method 3.

(10) Record the results on the data card enclosed with the sample.

(11) Evacuate the bag completely and repeat Steps 6 through 10.

Send the data card and the Tedlar bag to the address below. (The cylinder gas can should not be returned.)

Ms. Ellen Streib
Quality Assurance Division (MD-77)
Environmental Monitoring Systems Laboratory
U.S. Environmental Protection Agency
Research Triangle Park, NC 27711

NOTE: Site number will always be 001 except when other Orsat apparatus or participants are using the same gas sample. The extra apparatus or participants should be labeled 002, 003, etc.

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